

Form PTO-1390

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NO.

H 3266 PCT/US

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)

09/600913

INTERNATIONAL APPLICATION NO.
PCT/EP99/00198

INTERNATIONAL FILING DATE
January 15, 1999

PRIORITY DATE CLAIMED
January 24, 1998

TITLE OF INVENTION

**AUTOMATIC COMMAND AND CONTROL OF CLEANSING BATHS BY MEANS OF
ALKALINITY REGULATION**

APPLICANT(S) FOR DO/EO/US

Werner Opitz, Ludger Buetfering, Hans-Willi Kling, Ibolya Bartik-Himmeler, Wolfgang Krey and Stefan Nawrath

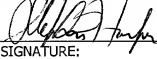
Applicant herewith submits to the United States Designated/Elected Office (EO/DO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
 2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
 3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
 4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
 5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
 6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
 7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
 8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
 9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (unexecuted)
 10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).
- Items 11. to 16. below concern other document(s) or information included:**
11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
 12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
 13. ☒ A **FIRST** preliminary amendment
 - ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
 14. ☐ A substitute specification.
 15. ☐ A change of power of attorney and/or address letter.
 16. ☒ Other items or information:

International Search Report (With Information Disclosure Citation and References)

"Express Mail" mailing label number EL 444424619US

U.S. Application No. (if known, see 37 CFR 1.5) 097600913	INTERNATIONAL APPLICATION NO. PCT/EP99/00198	ATTORNEY'S DOCKET NUMBER H 3266 PCT/US
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17. The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO..... \$970.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO..... \$840.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International search fee (37 CFR 1.445(a)(2)) paid to USPTO \$690.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4)..... \$96.00	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="text-align: left;">CALCULATIONS</th> <th style="text-align: left;">PCTO USE ONLY</th> </tr> <tr> <td>ENTER APPROPRIATE BASIC FEE AMOUNT =</td> <td>\$ 840</td> </tr> <tr> <td>Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).</td> <td>\$ 0</td> </tr> <tr> <td> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 20%;">CLAIMS</th> <th style="width: 20%;">NUMBER FILED</th> <th style="width: 20%;">NUMBER EXTRA</th> <th style="width: 20%;">RATE</th> <th style="width: 20%;"></th> <th style="width: 20%;"></th> </tr> <tr> <td>Total Claims</td> <td>1 - 20 =</td> <td>0</td> <td>0 X \$18.00</td> <td>\$ 0</td> <td></td> </tr> <tr> <td>Independent Claims</td> <td>1 - 3 =</td> <td>0</td> <td>0 X \$78.00</td> <td>\$ 0</td> <td></td> </tr> <tr> <td colspan="4">Multiple dependent claims (s)(if applicable) 0</td> <td>+ \$260.00</td> <td>\$ 0</td> </tr> <tr> <td colspan="4">TOTAL OF ABOVE CALCULATIONS</td> <td>=</td> <td>\$ 840</td> </tr> </table> </td> <td></td> </tr> <tr> <td>Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).</td> <td>\$ 0</td> </tr> <tr> <td>SUBTOTAL</td> <td>= \$ 840</td> </tr> <tr> <td>Processing fee of \$130.00 for furnishing the English translation later the <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).</td> <td>+ \$ 0</td> </tr> <tr> <td>TOTAL NATIONAL FEE</td> <td>= \$ 840</td> </tr> <tr> <td>Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property</td> <td>+ \$ 0</td> </tr> <tr> <td>TOTAL FEES ENCLOSED</td> <td>= \$ 840</td> </tr> <tr> <td></td> <td> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td>Amount to be refunded:</td> <td>\$-----</td> </tr> <tr> <td>charged:</td> <td>\$ 840.00</td> </tr> </table> </td> </tr> </table> <p>a. <input type="checkbox"/> A check in the amount of \$_____ to cover the above fees is enclosed.</p> <p>b. <input checked="" type="checkbox"/> Please charge my Deposit Account No. <u>01-1250</u> in the amount of \$840.00 to cover the above fees. A duplicate copy of this sheet is enclosed. Order No. <u>00-0762</u>.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>01-1250</u>. A duplicate copy of this sheet is enclosed.</p> <p>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.</p> <p>SEND ALL CORRESPONDENCE TO: Henkel Corporation, Law Dept. 2500 Renaissance Blvd., Suite 200 Gulph Mills, PA 19406</p> <p style="text-align: right;">  SIGNATURE: </p> <p style="text-align: right;"> Stephen D. Harper NAME ATTORNEY FOR APPLICANT 33,243 REGISTRATION NUMBER </p>	CALCULATIONS	PCTO USE ONLY	ENTER APPROPRIATE BASIC FEE AMOUNT =	\$ 840	Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	\$ 0	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 20%;">CLAIMS</th> <th style="width: 20%;">NUMBER FILED</th> <th style="width: 20%;">NUMBER EXTRA</th> <th style="width: 20%;">RATE</th> <th style="width: 20%;"></th> <th style="width: 20%;"></th> </tr> <tr> <td>Total Claims</td> <td>1 - 20 =</td> <td>0</td> <td>0 X \$18.00</td> <td>\$ 0</td> <td></td> </tr> <tr> <td>Independent Claims</td> <td>1 - 3 =</td> <td>0</td> <td>0 X \$78.00</td> <td>\$ 0</td> <td></td> </tr> <tr> <td colspan="4">Multiple dependent claims (s)(if applicable) 0</td> <td>+ \$260.00</td> <td>\$ 0</td> </tr> <tr> <td colspan="4">TOTAL OF ABOVE CALCULATIONS</td> <td>=</td> <td>\$ 840</td> </tr> </table>	CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE			Total Claims	1 - 20 =	0	0 X \$18.00	\$ 0		Independent Claims	1 - 3 =	0	0 X \$78.00	\$ 0		Multiple dependent claims (s)(if applicable) 0				+ \$260.00	\$ 0	TOTAL OF ABOVE CALCULATIONS				=	\$ 840		Reduction of 1/2 for filing by small entity, if applicable. 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24 JUL 2000

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PATENT

Docket No. H 3266 PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

RE: PCT/EP99/00198
International Filing Date: January 15, 1999
Priority Date Claimed: January 24, 1998
Applicant: Opitz et al.
Title: AUTOMATIC COMMAND AND CONTROL OF CLEANSING
BATHS BY MEANS OF ALKALINITY REGULATION
Applicants' Reference: H 3266 PCT/US

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Box PCT
Washington, DC 20231

ATTN: DO/EO/US

Sir:

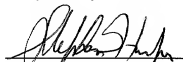
Before examining this application, please enter the following amendment.

IN THE CLAIMS:

Please cancel claims 2-14, without prejudice.

Should any fees be deemed necessary to enter this amendment, please charge
them to Deposit Account No. 01-1250.

Respectfully submitted,



Stephen D. Harper
(Reg. No. 33,243)
Attorney for Applicants
(610) 278-4927

Henkel Corporation
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2500 Renaissance Boulevard, Suite 200
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"Automatic checking and regulation of cleaner baths by determination of alkalinity"

This invention relates to a process for the automatic checking and regulation of cleaner baths, whereby the free alkalinity and/or the total alkalinity of the cleaner bath is determined by way of a measuring and regulating parameter and, where necessary, adjusted by metering measures. The process has been conceived in particular for technical cleaner baths in the metalworking industry, such as in automobile manufacture. It permits the functionality of the cleaner bath, characterised by the parameter "alkalinity", to be monitored automatically and, where necessary, enables the cleaner bath to be replenished automatically, or by external request, or enables other bath-maintenance measures to be initiated. In particular, the process is conceived in such a way that the results of the determinations are transmitted to a location remote from the cleaner bath. In addition, it is possible for interventions to be made in the automatic measuring sequence from a location remote from the cleaner bath or for make-up metering or other bath-maintenance measures to be initiated. The "location remote from the cleaner bath" may be situated, for example, in a higher-level process-control system, in a central control station of the plant in which the cleaner bath is located, or even outside the plant.

The cleaning of metallic parts prior to continued processing thereof constitutes a standard task in the metalworking industry. The metallic parts may, for example, be contaminated with pigment grime, dust, abraded metal, anti-corrosion oils, cooling lubricants or reshaping aids. Prior to continued processing, in particular prior to an anti-corrosion treatment (for example, phosphatisation, chromatisation, anodisation, reaction with complex fluorides, etc) or prior to lacquering, such contaminants have to be removed by a suitable cleaner solution. Spray processes, immersion processes or combined processes are considered for this purpose.

Industrial cleaners in the metalworking industry are, as a rule, alkaline (pH values in the range above 7, for example between 9 and 12). The basic constituents thereof are alkalis (alkali metal hydroxides, carbonates, silicates, phosphates, borates) and also non-ionic and/or anionic surfactants. The cleaners frequently contain, as additional auxiliary components, complexing agents (gluconates, polyphosphates, salts of amino carboxylic acids, such as ethylenediamine tetraacetate or nitrilotriacetate, salts of phosphonic acids, such as salts of

hydroxyethane diphosphonic acid, phosphonobutane tricarboxylic acid, or other phosphonic or phosphonocarboxylic acids), anti-corrosive agents, such as salts of carboxylic acids having 6 to 12 carbon atoms, alkanolamines and foam inhibitors, such as alkoxylates, closed at the terminal groups thereof, of alcohols having 6 to 16 carbon atoms in the alkyl residue.

5 Provided that the cleaner baths contain no anionic surfactants, cationic surfactants may also be used.

By way of non-ionic surfactants, the cleaners as a rule contain ethoxylates, propoxylates and/or ethoxylates/ propoxylates of alcohols or alkylamines having 6 to 16 carbon atoms in the alkyl residue, which may also be closed at the terminal groups thereof. By way of

0 anionic surfactants, alkyl sulfates and alkyl sulfonates are widely used. Alkyl benzene sulfonates are also encountered, but they are disadvantageous from the environmental point of view. By way of cationic surfactants, cationic alkylammonium compounds having at least one alkyl residue having eight or more carbon atoms are particularly preferred.

The alkalis in the cleaner bath contribute to the cleaning capacity thereof. For instance, they saponify saponifiable contaminants, such as fats and thereby make the latter soluble in water.

15 In addition, they contribute to the detachment of insoluble dirt from the surface of the metal by electrostatic repulsion, as a result of adsorption of OH ions rendering the surfaces negatively charged. As a result of reactions of this type, optionally also as a result of removal by entrainment, alkalinity is consumed, so that the cleaning effect diminishes with

0 time. It is therefore conventional to check the alkalinity of the cleaning baths at certain times and, where necessary, to replenish the solution with new active substances or to renew it entirely. This examination is carried out either manually or by means of an automatic titrator locally. In this connection the alkalinity is generally checked by titration with a strong acid.

25 The operating personnel assess the alkalinity on the basis of the acid consumption and take the necessary measures, such as bath replenishment or bath renewal. This currently conventional process assumes that operating personnel are present in the vicinity of the cleaning bath at the requisite checking times. The shorter the checking intervals that are desired, the greater are the demands made on the operating personnel for the check measurements.

From EP-A-806 244 a process is known for determining the pH of a solution automatically and for make-up metering of acid or lye automatically in the event of anomalies. The object thereof involves maintaining the pH of a stream of liquid at a predetermined value. Acid-base titration is not carried out using this process. In this case, it is necessary to check the functionality of the plant in situ. It is not possible to intervene from a remote location in the sequence of pH measurements and metering measures.

On the other hand, the present invention sets itself as an object the automating and documenting of the checking of cleaner baths by determination of the alkalinity in such a way that at least the results of the determination of alkalinity are stored on a data carrier and/or are output. The measuring device employed should preferably examine and calibrate itself and in the event of malfunction transmit an alarm signal to a remote point. Furthermore, it should preferably be possible to examine the functionality of the measuring device and the results of measurement from a remote point. Moreover, it should be possible for interventions to be made in the measuring sequence and in the bath-maintenance measures from a remote point. By virtue of the remote control desired, the effort expended by personnel on checking and regulation of the cleaner baths is to be reduced.

This object is achieved by means of a process for automatic determination of the alkalinity of one or more cleaning baths containing surfactant by acid-base reaction using an acid, whereby, subject to the use of a suitable measuring device under program control,

- (a) a sample of specified volume is drawn from a cleaning bath,
- (b) if desired, the sample is freed of solids,
- (c) a selection is made as to whether free alkalinity and/or total alkalinity is to be determined,
- (d) the sample is titrated by addition of an acid, or an acid is submitted and the latter is titrated with the sample,
- (e) the result of the titration is output and/or stored on a data carrier and/or utilised as the basis for further evaluations.

The sample volume drawn in step (a) may be permanently programmed into the control part of the measuring device to be employed for the process. The magnitude of the sample

volume may preferably be changed from a remote location. Furthermore, the control program may be so designed that it makes the sample volume to be used dependent on the result of a preceding measurement. For instance, the sample volume may be selected to be larger, the lower the alkalinity of the cleaner bath. The accuracy of the determination of alkalinity may be optimised by this means.

When reference is made to a "remote location", for the present purposes this is to be taken to mean a location that is not in direct contact, or at least not in optical contact, with the cleaner bath. The remote location may be, for example, a central process-control system that checks and regulates the cleaner bath as a sub-task within the context of an overall process for surface treatment of the metallic parts. The "remote location" may also be a central control and observation point from which the overall process is checked and regulated and which is located, for example, in a room differing from that of the cleaner bath. However, a point outside the plant in which the cleaner bath is located also comes into consideration by way of "remote location". By this means it becomes possible for specialists who remain outside the plant in which the cleaner bath is located to examine and regulate the cleaner bath. As a result, it is necessary for specialist personnel to be detained substantially less frequently at the location of the cleaner bath.

Suitable data lines with which the results of the determinations of alkalinity and also control commands may be transmitted are known.

Between the drawing of the sample and the actual measurement, it may be desirable to free the sample of solids in the optional step (b). In the case of a cleaner bath that is only slightly charged with solids, this is not necessary. However, in the case of a solids content of the cleaner bath that is too high, valves pertaining to the measuring device may become clogged and sensors, such as electrodes may be contaminated. It is therefore advisable to remove solids from the sample. This may be effected automatically by filtration or by using a cyclone separator or a centrifuge.

In step (c) a selection is made as to whether the free alkalinity and/or the total alkalinity is to be determined. This may be input permanently into the program control sequence. For

instance, both the free alkalinity and the total alkalinity may be determined in one determination cycle. However, the program may also decide to determine one of these two values more frequently than the other. This may be the case, for example, when determinations carried out previously have shown that one of the two values changes more rapidly than the other. Of course, the choice of whether free alkalinity or total alkalinity is to be determined may also be made by means of an external request. For the present purposes, the expression "external request" is to be understood to mean that it is possible for interventions to be made in the automated determination sequence either by a higher-level process-control system or manually via a data line.

The terms "free alkalinity" and "total alkalinity" are not unambiguously defined and are handled differently by the various users. For instance, particular pH values may be defined, up to which titration has to be effected in order to determine either the free alkalinity or the total alkalinity, for example $\text{pH} = 8$ for free alkalinity, $\text{pH} = 4.5$ for total alkalinity. These preselected pH values have to be input into the control system for the automatic determination process. As an alternative to particular pH values, the transition points of certain indicators may also be selected with a view to establishing the free alkalinity and the total alkalinity. Alternatively, turning-points in the curve of pH values may be selected and defined as end points for the free alkalinity or the total alkalinity.

With a view to actual determination of the alkalinity in step (d), use is made of the acid-base reaction with an acid. A strong acid is preferably selected for this purpose. In this connection, the sample may be titrated by addition of an acid until the specified criteria either for free alkalinity or for total alkalinity are attained. Alternatively, the acid may be submitted and titrated with the sample.

The result of the titration is subsequently output and/or stored on a data carrier (step (e)). In this connection, the data carrier may be situated at the location of the determination or in a remote arithmetic logic unit. The expression "output of the result of the titration" is to be understood to mean that the result is either passed on to a higher-level process-control system or is displayed on a screen or printed out in a recognisable manner. In this connection, the location of the display or the output of the result may be the "remote location" defined

above. Preferably, the results of the individual determinations are stored on a data carrier at least for a specified time interval, so that they may subsequently be evaluated, in the sense of quality assurance, for example. However, the results of the determinations of alkalinity do not have to be output or stored on data carriers immediately. Instead, they may also be utilised directly as the basis for further calculations, the results of these further calculations being displayed or stored. For instance, instead of the current alkalinity value in each case, the trend of the alkalinity values and/or the relative change thereof may be displayed. Alternatively, the current alkalinity values may be converted into "% of nominal content".

In the simplest case, the process according to the present invention operates in such a way that steps (a) to (e) are repeated after a specified time interval. This specified time interval depends on the requests of the operator of the cleaning bath and may comprise any time interval in the range from about five minutes to several days. For quality assurance it is preferable that the specified time intervals lie, for example, in the range between five minutes and two hours. For instance, a measurement may be carried out every 15 minutes.

However, the process according to the present invention may also be implemented in such a way that steps (a) to (e) are repeated after time intervals that are shorter, the more the results of two consecutive determinations differ. The control system for the process according to the present invention may also decide itself whether the time intervals between the individual determinations are to be shortened or lengthened. Of course, the control system has to be provided in advance with an instruction as to which time intervals are to be selected for which differences between the results of consecutive determinations.

Moreover, the process according to the present invention may be implemented in such a way that steps (a) to (e) may be carried out at any time by means of an external request. As a result, immediate checking, for example of the alkalinity content of the cleaner bath, may be undertaken if quality problems are established in subsequent process steps. Measurement of the alkalinity may also take place in a time-controlled manner (in accordance with fixed time intervals) or in an event-controlled manner (in the event of established changes or as a result of external requests).

5 The process according to the present invention is preferably implemented in such a way that the measuring device employed checks itself and, where necessary, recalibrates itself. To this end, provision may be made such that, after a specified time interval or after a specified number of determinations or by reason of an external request, the functionality of the measuring device employed is examined by check measurements of one or more standard solutions. For the purpose of examination, a standard solution having known contents of free and total alkalinity is titrated. This examination is closest to reality if a standard cleaner solution is employed by way of standard solution, the composition of which approximates as closely as possible to the cleaner solution to be examined. The standard solutions are preferably maintained subject to exclusion of air or under a protective gas (nitrogen, for example).

10 A central issue when examining the functionality of the measuring device is constituted by the checking of the sensor employed. For instance, the latter may be a pH-sensitive electrode, in particular a glass electrode. With the aid of a buffer solution by way of standard solution, it is possible to examine whether the electrode is supplying the expected voltage, whether it responds within the expected time and whether the slope thereof (= change in voltage as a function of the change in pH) lies within the nominal range. If this is not the case, the measuring device outputs an alarm signal locally or, preferably, at a remote location. This alarm signal may contain a suggestion for intervention that is selected by the control program of the measuring device or by the higher-level process-control system. For instance, it may be suggested that the electrode be cleaned or exchanged.

15 In the process according to the present invention there may also be provision such that the functionality of the measuring device employed is examined by check measurement of one or more standard solutions if the results of two consecutive measurements differ by a specified amount. By this means it is possible to distinguish whether established anomalies in the alkalinity of the cleaner bath are real and require bath-maintenance measures or whether they are being simulated by an error in the measuring system.

25 Depending on the result of the examination of the measuring device employed, the determinations of alkalinity that have taken place between the current check measurement and

the preceding one may be provided with a status identifier that denotes the reliability of these determinations of alkalinity. If, for example, consecutive check measurements for the purpose of examining the measuring device employed have shown that the latter is operating correctly, the determinations of the alkalinity may be provided with a status identifier "OK".

If the results of the check measurements differ by a specified minimum amount, the determinations of the alkalinity that have taken place in the meantime may, for example, be provided with the status identifier "dubious".

Furthermore, provision may be made such that, depending on the result of the examination of the measuring device employed, automatic determination of the alkalinity is continued and/or one or more of the following actions are carried out: analysis of established anomalies, correction of the measuring device, termination of the determination of alkalinity, transmission of a status signal or an alarm signal to a higher-level process-control system or to a monitoring device - that is to say, to a remote location. Accordingly the measuring device may, if desired, decide, in accordance with specified criteria, whether it is operational to such an extent that the determinations of alkalinity may be continued or whether anomalies are established that necessitate manual intervention.

Various sensors are suitable for tracking the acid-base reaction of the cleaner solution with the acid employed for the titration. In accordance with the present state of the art, use will preferably be made of a pH-sensitive electrode, such as a glass electrode. The latter supplies a pH-dependent voltage signal which may be evaluated further. The use of an electrode of this type is particularly straightforward in terms of apparatus and is therefore preferred.

However, with a view to tracking the acid-base reaction of step (d), use may also be made of an indicator, the pH-dependent interaction of which with electromagnetic radiation is measured. For instance, this indicator may be a classical color indicator, the change in color of which is measured photometrically. Alternatively, use may be made of an optical sensor. In this connection it is, for example, a layer of an inorganic or organic polymer having a fixed dyestuff that changes color at a certain pH. The change in color is based, as in the case of a classical color indicator, on the fact that hydrogen ions or hydroxide ions that are able to diffuse into the layer react with the dyestuff molecules. The change in the optical

properties of the layer may be determined photometrically. Alternatively, films, such as organic polymers, may be used, the refractive index of which changes as a function of the pH. If, for example, a light guide is coated with such a polymer, it is possible to ensure that total reflection occurs in the light guide on one side of a threshold value of the refractive index, so that a ray of light is transmitted. On the other side of the threshold value of the refractive index, however, total reflection no longer occurs, so that the ray of light exits the light guide. At the end of the light guide it is then possible to detect whether the light is being propagated by the light guide or not. A device of this type is known as an "optrode".

Furthermore, inorganic or organic solids, the electrical properties of which change with the pH of the surrounding solution, may be employed as sensors. For instance, use may be made of an ion conductor, the conductivity of which depends on the concentration of the H^+ or OH^- ions. By measurement of the d.c. or a.c. conductivity of the sensor, it is then possible for the pH of the surrounding medium to be inferred.

The measuring system employed in the process according to the present invention is preferably designed such that it automatically monitors the filling levels and/or the consumption of the reagents employed (acids, standard solutions and test solutions, possibly auxiliary solutions) and, in the event of a specified minimum filling level not being attained, outputs a warning signal. By this means it is possible to prevent the measuring device from becoming non-functional as a result of lacking the necessary chemicals. Monitoring of the filling levels may be effected using known methods. For instance, the vessels with the chemicals may be situated on a balance that registers the respective weight of the chemicals. Or use may be made of a float. Alternatively, a minimum filling level may be examined by means of a conductivity electrode that is immersed in the vessel containing the chemical. The warning signal to be output by the measuring device is preferably transmitted to the remote location, so that the appropriate measures may be initiated from there. In general, provision is preferably made in the process according to the present invention such that the results of the determinations and/or of the check measurements and/or of the calibrations and/or the status signals are transmitted to a remote location continuously or at specified time intervals and/or on request. By this means, monitoring personnel that do not have to be present at the location of the cleaner bath are kept constantly informed of the current

alkalinity content thereof. Depending on the results of the determinations and of the check measurements, necessary corrective measures may be taken either automatically via a process-guidance system or as a result of manual intervention.

5 In the event of a specified minimum value of the alkalinity (free and/or total alkalinity) not being attained or on external request, the simplest corrective measure involves a device being activated that meters one or more replenishing components (solution or powder) into the cleaning bath. This may be effected, for example, in an automated manner in such a way that, depending on the alkalinity content ascertained, a certain quantity of replenishing solution or replenishing powder is supplied to the cleaning bath. In this regard, the magnitude of the added portion itself or, in the case of firmly specified added portions, the time intervals between the individual additions may be varied. This may be effected, for example, with the aid of metering pumps or in a weight-controlled manner. In the process according to the present invention there is provision, on the one hand, such that in the event of certain deviations from the nominal value (particularly if the functionality of the measuring device is established by the check measurements), a certain quantity of replenishing component is metered into the cleaning bath in order to make it up. On the other hand, however, this make-up metering may also be undertaken by reason of an external request, from a remote location for example, irrespective of the current alkalinity content.

10 In another embodiment of the present invention the cleaning bath is replenished in a throughput-dependent manner using a specified quantity of replenishing component per unit passed through (basic metering). For instance, in the case of a cleaning bath for automobile bodies the quantity of replenishing component that is added per cleaned body may be established. The checking, in accordance with the present invention, of the alkalinity then serves to check and to document the success of this specified addition and also, by means of additional event-dependent fine metering (additional metering in the event of the nominal values not being attained, suspension of the basic metering in the event of the nominal values being exceeded), to achieve a more constant mode of operation of the cleaning bath. By this means, fluctuations in quality are reduced.

25 Of course, the process according to the present invention assumes that the appropriate device

is made available. The latter includes a control system, preferably a computer control system, which controls the course of measuring in a time-dependent and/or event-dependent manner. It must furthermore include the necessary reagent vessels, pipelines, valves, metering and measuring devices etc for regulation and measurement of the sample streams.

- 5 The materials should be adapted to the intended use, for example they should consist of high-grade steel and/or synthetic material. The control electronics of the measuring device should comprise an appropriate input-output interface, in order to be able to communicate with a remote location.

- 0 The process according to the present invention makes it possible, on the one hand, to check the alkalinity of cleaning baths in situ and to initiate specified corrective measures without manual intervention. By this means the process safety is enhanced and a constantly reliable cleaning result is achieved. Deviations from the nominal values may be detected at an early stage and corrected automatically or manually before the cleaning result is impaired. On the other hand, the measured data are preferably transmitted to a remote location, so that
15 operating or supervising personnel are kept constantly informed of the state of the cleaning bath also when it is not located in the immediate vicinity thereof. The effort expended by personnel on checking and regulation of the cleaning bath may be considerably reduced by this means. By virtue of the documentation of the data collected in the process according to the present invention, the requirements of modern quality assurance are taken into account.
20 The consumption of chemicals may be documented and optimised.

Claims:

1. A process for automatic determination of the alkalinity of one or more cleaning baths containing surfactant by acid-base reaction with an acid, whereby, subject to the use of a suitable measuring device under program control,
 - (a) a sample of specified volume is drawn from a cleaning bath,
 - (b) if desired, the sample is freed of solids,
 - (c) a selection is made as to whether free alkalinity and/or total alkalinity is to be determined,
 - (d) the sample is titrated by addition of an acid, or an acid is submitted and the latter is titrated with the sample,
 - (e) the result of the titration is output and/or stored on a data carrier and/or utilised as the basis for further evaluations.
2. A process as claimed in claim 1 wherein steps (a) to (e) are repeated after a specified time interval.
3. A process as claimed in claim 1 wherein steps (a) to (e) are repeated after time intervals that are shorter, the more the results of two consecutive determinations differ.
4. A process as claimed in claim 1 wherein steps (a) to (e) are carried out by reason of an external request.
5. A process as claimed in one or more of claims 1 to 4 wherein, after a specified time interval or after a specified number of determinations or by reason of an external request, the functionality of the measuring device employed is examined by check measurement of one or more standard solutions.
6. A process as claimed in one or more of claims 1 to 4 wherein the functionality of the measuring device employed is examined by check measurement of one or more

standard solutions if the results of two consecutive measurements differ by a specified amount.

7. A process as claimed in one or both of claims 5 and 6 wherein, depending on the result of the examination of the measuring device employed, the determinations of alkalinity that have taken place between the current check measurement and the preceding one are provided with a status identifier that denotes the reliability of these determinations of alkalinity.
8. A process as claimed in one or both of claims 5 and 6 wherein, depending on the result of the examination of the measuring device employed, automatic determination of the alkalinity is continued and/or one or more of the following actions are carried out: analysis of established anomalies, correction of the measuring device, termination of the determinations of alkalinity, transmission of a status signal or an alarm signal to a higher-level process-control system or to a monitoring device.
9. A process as claimed in one or more of claims 1 to 8 wherein, when step (d) is implemented, a pH-sensitive electrode is employed.
10. A process as claimed in one or more of claims 1 to 8 wherein, when step (d) is implemented, an indicator is employed, of which the pH-dependent interaction with electromagnetic radiation is measured.
11. A process as claimed in one or more of claims 1 to 8 wherein, when step (d) is implemented, use is made of a substance, the color and/or refractive index and/or electrical conductivity of which changes with the pH value of the surrounding solution.
12. A process as claimed in one or more of claims 1 to 11 wherein the filling levels of the reagents employed are automatically monitored and a warning signal is output in the event of a specified minimum filling level not being attained.

13. A process as claimed in one or more of claims 1 to 12 wherein the results of the determinations and/or of the check measurements and/or of the calibrations and/or the status signals are transmitted continuously or at specified time intervals and/or on request to a location differing from the location of the determination.

5 14. A process as claimed in one or more of claims 1 to 13 wherein, in the event of a specified minimum value of the alkalinity not being attained or on request, a device is activated that meters one or more replenishing components into the cleaning bath.

0050060-21600960

Abstract

"Automatic checking and regulation of cleaner baths by determination of alkalinity"

A process for automatic determination of the alkalinity of cleaning baths by acid-base reaction, whereby the determination is effected in automated manner and under program control and the results of the determination may be transmitted to a remote location. From there, it is also possible for interventions to be made during the course of the determinations. Bath-maintenance measures may be initiated automatically in accordance with specified criteria or as a result of a request from a remote location. The process reduces the effort exerted by personnel on bath checking and bath maintenance and enhances the process safety.

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DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

☐ Declaration Submitted with Initial Filing OR ☒ Declaration Submitted after Initial Filing

Attorney Docket Number H 3266 PCT/US

First Named Inventor Opitz, Werner

COMPLETE IF KNOWN

Application Number

Filing Date

Group Art Unit

Examiner Name

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

AUTOMATIC COMMAND AND CONTROL OF CLEANSING BATHS BY MEANS OF ALKALINITY REGULATION

(Title of the invention)

the specification of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) 1/15/1999 as United States Application Number or PCT International

Application Number PCT/EP98/00198 and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, § 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
198 02 725.7	Germany	1/24/1998	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
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			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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☐ Additional foreign application numbers are listed on a supplemental priority sheet attached hereto:

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	Additional provisional application numbers are listed on a supplemental priority sheet attached hereto.
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DECLARATION

Page 2

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365 of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)
	PCT/EP99/00198	1/15/1999	

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority sheet attached hereto.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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005060-01000360

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